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Adsorption near Ambient Temperatures of Methane, Carbon Tetrafluoride, and Sulfur Hexafluoride on Commercial Activated Carbons

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The adsorption isotherms for CH_4, CF_4, and SF_6 are measured at three or four temperatures near ambient on three commercial activated carbons. The data are reduced using a virial-type equation of adsorption. Using this equation, isosteric heats of adsorption are calculated. It is shown that this fundamental thermodynamic quantity provides a basis for differentiating between the carbons' micropore structures.

Introduction

Gas adsorption characteristics of activated carbons are strongly dependent on their microstructure. An indication of the importance of this property is that the specific surface area and pore size distribution are proposed as fundamental quantities recommended by IUPAC to characterize these materials. Depending upon their application, carbons with different pore structures are preferred. In particular, for gas separation or natural gas and hydrogen storage, microporous carbons are desired. Micropores play the most important role in the adsorption of small molecules at temperatures above their boiling point at moderate pressures due to the enhancement of the adsorption potential in such pores (Everett and Powl, 1976).

The data presented in this paper were obtained as a part of a continuing study of the relationship between the micropore structure of carbons and their adsorption properties (Jagiełło and Schwarz, 1993; Jagiełło *et al.*, 1995). In our studies we have chosen symmetrical adsorbates with different molecular sizes that interact nonspecifically with carbon surfaces. The results we obtain are useful for comparison with theoretical modeling of adsorption in microporous carbons.

Experimental Section

Materials. Three activated carbons from different sources were analyzed in this study: molecular sieving carbon Carbosieve G (G) (Suppelco), Maxsorb (M) (Kansai Coke and Chemicals Inc.), and Westvaco activated carbon (W).

Methods. Volumetric Sorption Experiments. Nitrogen isotherms were measured by a GEMINI III 2375 surface area analyzer (Micromeritics) at 77 K. Before the experiment, the samples were heated for 10 h at 473 K and then outgassed at this temperature under a vacuum of 10^{-3} kPa. The isotherms were used to calculate the specific surface area and pore volume.

Methane, carbon tetrafluoride, and sulfur hexafluoride adsorption isotherms were also measured with the same equipment using the same pretreatment of samples as in the case of nitrogen adsorption. The isotherms were measured around room temperature (see Tables 2−4). The apparatus was equipped with a home-made thermostated system controlled by a Fisher Scientific isotemp refrigerated circulator, model 900.

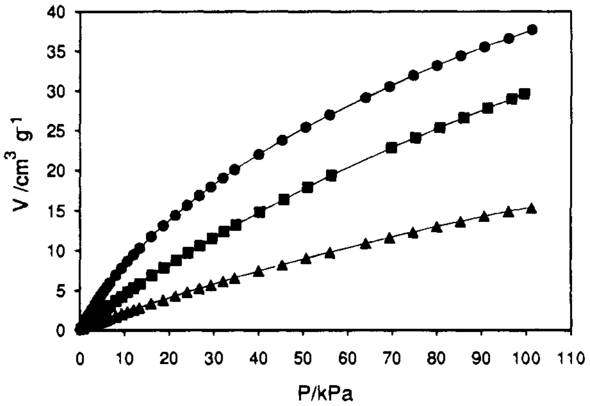

Figure 1. Adsorption isotherms of CF_4 at 296 K (●, G; ■, M) and 297 K (▲, W).

Table 1. Structural Parameters Calculated from Nitrogen Adsorption Isotherms

sample	S_{BET}/(m^2·g^{-1})	S_L/(m^2·g^{-1})	V_{mic}/(cm^3·g^{-1})
Carbosieve G	1100	1460	0.560
Westvaco	1160	1700	0.470
Maxsorb	1630	2300	0.760

Results and Discussion

The structural parameters calculated from nitrogen adsorption isotherms are collected in Table 1. The specific surface areas were calculated assuming both the BET and Langmuir adsorption models; micropore volumes, V_{mic}, were calculated using the Dubinin−Radushkevich method (Dubinin, 1966). These quantities are considered here as standard parameters used to describe the sorption capacity of our materials. Table 1 shows that the carbons selected for this study are representative of a broad range of microstructural properties.

The raw data for the adsorption isotherms of CH_4, CF_4, and SF_6, measured at different temperatures are collected in Tables 2−4. To illustrate the variation in adsorption isotherms for each carbon that relates to their different porous structure, we compare in Figure 1 the isotherms obtained for CF_4 at similar temperatures (296 and 297 K).

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Table 2. Experimental Results on Carbosieve G Molecular Sieving Carbon (Volumes of Gas Refer to p = 101.325 kPa and T = 273.15 K)

	$v_{CH_4}/(cm^3 \cdot g^{-1})$			$v_{CF_4}/(cm^3 \cdot g^{-1})$			$v_{SF_6}/(cm^3 \cdot g^{-1})$			
P/kPa	258 K	275 K	295 K	258 K	275 K	296 K	267 K	275 K	282.5 K	296 K
0.133				0.87	0.39	0.13	10.93	6.68	5.42	1.90
0.267	0.55	0.19		1.57	0.65	0.16	15.18	10.98	8.60	4.14
0.547	1.30	0.63	0.30	3.12	1.50	0.56	22.72	16.97	13.45	7.56
0.800	1.77	0.85	0.33	4.26	2.10	0.77	26.86	21.08	16.94	9.97
1.07	2.40	1.23	0.55	5.43	2.79	1.12	29.82	24.17	19.68	12.04
1.33	2.87	1.47	0.62	6.43	3.35	1.34	32.62	27.11	22.07	13.90
1.61	3.49	1.86	0.86	7.49	4.02	1.69	35.10	29.45	24.15	15.56
2.00	4.14	2.21	1.00	8.75	4.77	2.01	38.29	32.40	26.91	17.73
2.67	5.35	2.93	1.38	10.82	6.08	2.67	42.64	36.67	30.74	20.96
3.33	6.44	3.61	1.74	12.64	7.27	3.26	46.10	40.05	34.35	23.64
4.00	7.50	4.26	2.09	14.32	8.39	3.85	49.01	42.95	36.97	26.01
4.68	8.50	4.89	2.43	15.85	9.44	4.41	51.47	45.43	39.14	28.10
5.33	9.42	5.47	2.75	17.24	10.39	4.93	53.59	47.55	41.18	29.93
6.00	10.34	6.06	3.09	18.59	11.34	5.46	55.49	49.51	42.97	31.65
6.67	11.22	6.63	3.41	19.84	12.23	5.95	57.15	51.24	44.70	33.20
8.00	12.91	7.78	4.11	22.13	13.88	6.92	60.00	54.20	47.53	35.93
9.33	14.44	8.82	4.71	24.21	15.42	7.82	63.13	56.72	50.12	38.31
10.67	15.91	9.81	5.30	26.14	16.85	8.68	65.12	58.87	51.98	40.42
12.00	17.28	10.76	5.87	27.90	18.20	9.51	66.90	60.72	53.47	42.32
13.33	18.60	11.68	6.43	29.56	19.47	10.29	68.62	62.33	55.04	44.03
16.00	21.05	13.42	7.51	32.55	21.80	11.77	70.89	65.05	57.83	47.00
18.67	23.30	15.04	8.55	35.22	23.93	13.15	72.72	67.27	60.18	49.52
21.33	25.41	16.59	9.55	37.64	25.89	14.45	74.18	69.13	62.16	51.70
24.00	27.38	18.05	10.50	39.84	27.69	15.69	75.57	70.72	63.89	53.61
26.68	29.25	19.45	11.43	41.89	29.40	16.86	76.78	72.12	65.40	55.32
29.34	31.01	20.78	12.33	43.78	30.99	17.99	77.99	73.33	66.74	56.83
32.01	32.68	22.06	13.20	45.53	32.48	19.06	78.96	74.41	67.93	58.19
34.68	34.27	23.29	14.04	47.17	33.89	20.07	79.93	75.37	69.01	59.43
40.01	37.26	25.61	15.66	50.17	36.50	21.99	81.49	77.03	70.87	61.59
45.36	40.01	27.79	17.20	52.87	38.88	23.76	82.80	78.44	73.05	63.43
50.69	42.57	29.83	18.66	55.30	41.05	25.42	83.83	79.65	74.43	65.02
56.01	44.96	31.76	20.07	57.52	43.06	26.99	84.73	80.70	75.51	66.41
64.02	48.29	34.47	22.11	60.52	45.81	29.18	85.90	82.06	77.10	68.22
69.35	50.35	36.16	23.39	62.33	47.51	30.58	86.56	82.85	78.07	69.27
74.69	52.30	37.79	24.63	64.02	49.10	31.93	87.15	83.57	78.89	70.23
80.03	54.16	39.34	25.82	65.59	50.59	33.21	87.72	84.23	79.52	71.10
85.37	55.92	40.84	26.97	67.06	52.00	34.42	88.26	84.83	80.26	71.90
90.69	57.62	42.27	28.10	68.44	53.34	35.54		85.38		72.62
96.03	59.25	43.67	29.18	69.75	54.61	36.64		85.88		73.30
101.26	60.78	44.98	30.20	70.96	55.80	37.70		86.34		73.91

Considering the characteristics given in Table 1, the amount of adsorbed CF_4 is related neither to the carbon surface area nor to its micropore volume. The highest uptake in the case of G carbon indicates its high content of small micropores which are mainly responsible for the adsorption at supercritical temperatures. The amount adsorbed for CH_4 and SF_6 follows the same trend as observed for CF_4.

In addition to the tabulated raw data, we report the parameters of the virial-type equation which was used for their correlation. We applied the following form of a virial-type equation:

$$\ln p = \ln v + (1/T)\sum_{i=0}^{m} a_i v^i + \sum_{i=0}^{n} b_i v^i \qquad (1)$$

where v, p, and T are the amount adsorbed, pressure, and temperature, respectively and a_i and b_i are empirical parameters. This equation was derived (Czepirski and Jagiełło, 1989) under the assumption that in the limited range of temperatures the isosteric enthalpy of adsorption, Q_{st}, is temperature invariant and that the adsorption isotherms obey Henry's law in the limit of zero pressure.

There is also known another formulation of the virial-type equation (Avgul and Kiselev, 1970):

$$\ln p = \ln v + \sum_{i=0}^{M} C_i v^i \qquad (2)$$

where it is assumed that each C_i coefficient is expressed by

$$C_i = a_i/T + b_i \qquad (3)$$

Equations 1 and 2 become equivalent when both polynomials in eq 1 are of the same order, i.e., $n = m = M$. It was shown (Czepirski and Jagiełło, 1989; Bandosz *et al.*, 1989, Jagiełło *et al.*, 1992; Bandosz *et al.*, 1993) that eq 1 can describe, within experimental accuracy, different adsorption data using relatively low polynomial orders and that usually $n < m$.

We fit eq 1 to our results using a least squares procedure which minimizes the weighted sum of squared residuals:

$$S = \sum_{i=1}^{N} [w_i(\ln p_i^{\text{exptl}} - \ln p_i^{\text{eq}})]^2 \qquad (4)$$

where w_i are the statistical weights and superscripts exptl and eq refer to the experimental and calculated pressures, respectively. Statistical weights should reflect our knowledge about the experimental error. In our case, on the basis of the method of measurement, we assume a constant error in the measured v values. In order to correctly account for this error, w_i is calculated as a function of v:

$$w(v) = \left(\frac{\partial v}{\partial \ln p}\right)_T \qquad (5)$$

Table 3. Experimental Results on Westvaco Carbon (Volumes of Gas Refer to p = 101.325 kPa and T = 273.15 K)

	$v_{CH_4}/(cm^3 \cdot g^{-1})$			$v_{CF_4}/(cm^3 \cdot g^{-1})$		$v_{SF_6}/(cm^3 \cdot g^{-1})$			$v_{CF_4}/(cm^3 \cdot g^{-1})$	
P/kPa	268 K	283 K	298 K	283 K	297 K	268 K	283 K	298 K	P/kPa	268 K
0.133						1.81	0.97	0.48	0.520	0.31
0.267						2.92	1.59	0.79	0.707	0.38
0.533						4.81	2.81	1.53	0.880	0.55
0.800						6.35	3.81	2.17	1.05	0.61
1.07				0.22		7.64	4.69	2.75	1.31	0.85
1.33	0.50			0.30		8.81	5.48	3.28	1.76	1.09
1.60	0.54	0.30		0.39	0.26	9.85	6.21	3.77	2.19	1.33
2.00	0.68	0.39	0.21	0.54	0.38	11.34	7.25	4.50	2.63	1.57
2.67	1.02	0.63	0.41	0.81	0.60	13.46	8.78	5.62	3.08	1.81
3.33	1.32	0.84	0.55	1.01	0.76	15.34	10.13	6.58	3.51	2.04
4.00	1.57	1.03	0.69	1.21	0.91	17.04	11.36	7.45	3.95	2.27
4.67	1.80	1.18	0.80	1.41	1.06	18.58	12.48	8.27	4.39	2.49
5.33	2.02	1.33	0.91	1.59	1.20	20.01	13.54	9.05	5.27	2.92
6.00	2.24	1.49	1.01	1.78	1.35	21.37	14.54	9.77	6.15	3.34
6.67	2.46	1.63	1.11	1.96	1.49	22.63	15.47	10.47	7.03	3.75
8.00	2.88	1.92	1.31	2.32	1.76	25.00	17.24	11.78	7.89	4.13
9.33	3.29	2.19	1.51	2.67	2.04	27.16	18.87	13.01	8.77	4.51
10.67	3.69	2.47	1.69	3.02	2.31	29.16	20.37	14.16	10.52	5.25
12.00	4.08	2.74	1.88	3.36	2.57	31.00	21.78	15.24	12.28	5.95
13.35	4.46	2.99	2.05	3.70	2.83	32.74	23.12	16.27	14.03	6.62
16.00	5.18	3.51	2.43	4.35	3.34	35.90	25.60	18.15	15.79	7.28
18.67	5.90	4.01	2.79	4.99	3.83	38.76	27.86	19.91	17.55	7.93
21.33	6.57	4.49	3.14	5.60	4.32	41.35	29.94	21.53	19.31	8.54
24.00	7.23	4.95	3.48	6.20	4.79	43.74	31.89	23.05	21.05	9.14
26.68	7.85	5.41	3.83	6.79	5.25	45.96	33.72	24.50	22.81	9.73
29.34	8.46	5.86	4.15	7.36	5.71	48.03	35.44	25.87	26.30	10.85
32.01	9.06	6.29	4.49	7.91	6.14	49.94	37.06	27.17	29.82	11.93
34.66	9.65	6.73	4.79	8.45	6.58	51.74	38.59	28.40	33.33	12.96
40.00	10.77	7.55	5.43	9.51	7.43	55.05	41.44	30.74	36.84	13.96
45.34	11.85	8.35	6.02	10.53	8.25	58.04	44.05	32.90	42.10	15.38
50.68	12.87	9.12	6.62	11.51	9.04		46.45	34.91	45.62	16.29
56.01	13.87	9.87	7.18	12.46	9.81		48.68	36.80	49.13	17.17
64.02	15.29	10.96	8.00	13.83	10.93		51.73	39.42	52.64	18.04
69.35	16.20	11.64	8.54	14.70	11.64			41.04	56.16	18.87
74.69	17.09	12.34	9.08	15.56	12.34			42.56	59.65	19.69
80.03	17.96	13.00	9.61	16.39	13.02			43.92	63.17	20.48
85.37	18.81	13.65	10.12	17.21	13.69				65.34	20.96
90.70	19.62	14.30	10.62	18.00	14.35					
96.03	20.42	14.92	11.11	18.77	14.99					
101.17	21.18	15.52	11.58	19.24	15.38					

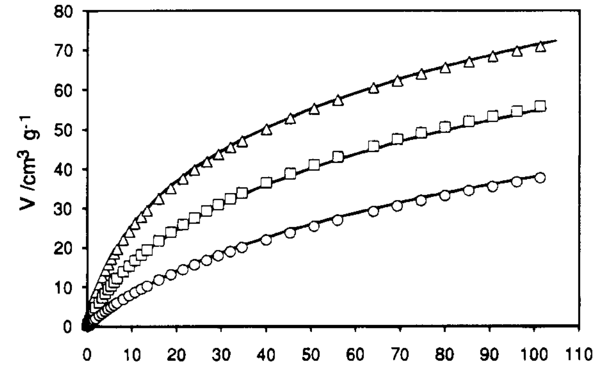

Figure 2. Adsorption isotherms of CF_4 on Carbosieve G measured at (△) 258 K, (□) 275 K, and (○) 296 K together with the isotherms calculated from eq 1 (solid lines).

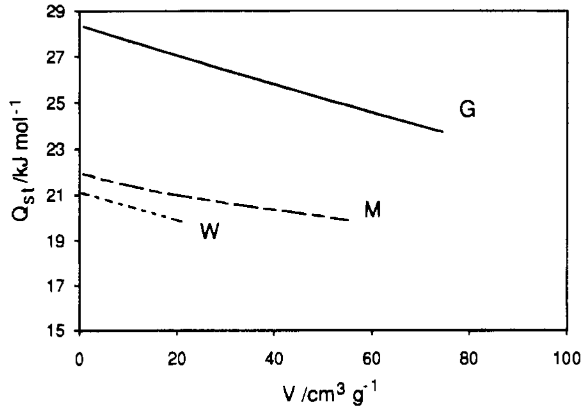

Figure 3. Isosteric enthalpies of CF_4 adsorption on the carbons studied.

Since the function $\ln p$ vs v is not known a priori, the fitting is performed iteratively. Assuming all $w_i = 1$, an initial fit is obtained and then eq 5 is applied. Usually after two steps this process converges. In order to establish optimal orders of polynomials, n and m, for our results we have analyzed the rms error of fit, σ,

$$\sigma = \sqrt{S/N} \tag{6}$$

as a function of n and m. Plots of this function were discussed elsewhere (Jagiełło *et al.*, 1992). The smallest values for n and m were chosen for which the fits did not significantly improve. In all cases satisfactory fits were obtained for $m \leq 3$ and $n = 0$. The fitting parameters and rms errors, σ, are listed in Table 5; σ describes the error in v values. An example of the fit is given in Figure 2 where the adsorption isotherms of CF_4 on Carbosieve G at three different temperatures are presented. It is seen that the fit is very good, although according to Table 5 only four parameters were used in this case.

The knowledge of the temperature dependence of adsorption enables one to study the thermodynamic properties of the adsorption system. The isosteric enthalpy of adsorption, Q_{st}, which is the fundamental thermodynamic

Table 4. Experimental Results on Maxsorb Carbon (Volumes of Gas Refer to p = 101.325 kPa and T = 273.15 K)

	v_{CH_4}/(cm^3·g^{-1})			v_{CF_4}/(cm^3·g^{-1})			v_{SF_6}/(cm^3·g^{-1})		
P/kPa	268 K	286 K	296 K	268 K	283 K	296 K	268 K	273 K	283 K
0.120							3.40	3.00	1.75
0.280				0.39	0.22	0.12	6.02	5.26	3.38
0.560				0.67	0.33	0.16	9.88	8.74	5.73
0.867	0.52			1.01	0.53	0.27	13.15	11.62	7.82
1.12	0.55			1.46	0.84	0.52	15.73	13.95	9.49
1.36	0.85	0.38		1.73	0.98	0.58	18.30	16.23	11.31
1.68	0.93	0.37		2.02	1.13	0.67	20.62	18.30	12.90
2.03	1.36	0.48		2.48	1.42	0.87	23.62	21.00	14.99
2.72	1.94	0.83	0.50	3.43	2.00	1.28	28.41	25.27	18.38
3.47	2.15	1.09	0.72	4.11	2.55	1.69	32.45	28.91	21.25
4.09	2.48	1.37	0.92	4.82	2.97	1.98	36.19	32.30	23.91
4.79	2.86	1.63	1.10	5.49	3.42	2.28	39.63	35.45	26.36
5.43	3.19	1.86	1.28	6.13	3.84	2.56	42.74	38.27	28.62
6.08	3.55	2.09	1.46	6.76	4.27	2.86	45.73	41.00	30.80
6.79	3.93	2.30	1.63	7.38	4.68	3.06	48.49	43.53	32.83
8.11	4.65	2.82	1.99	8.57	5.48	3.70	53.63	48.25	36.68
9.41	5.34	3.35	2.33	9.68	6.26	4.24	58.23	52.50	40.15
10.77	6.07	3.82	2.69	10.80	7.02	4.79	62.52	56.47	43.42
12.12	6.74	4.24	3.03	11.85	7.75	5.32	66.41	60.13	46.44
13.48	7.43	4.71	3.36	12.89	8.49	5.85	70.07	63.59	49.36
16.13	8.75	5.65	4.05	14.83	9.87	6.87	76.52	69.73	54.59
18.84	10.05	6.58	4.71	16.68	11.20	7.86	82.24	75.21	59.36
21.53	11.27	7.62	5.37	18.43	12.48	8.82	87.31	80.16	63.74
24.20	12.44	8.53	6.01	20.09	13.70	9.73	91.83	84.58	67.70
26.89	13.60	9.39	6.63	21.72	14.90	10.64	95.95	88.65	71.40
29.61	14.74	10.55	7.26	23.28	16.06	11.52	99.70	92.36	74.80
32.28	15.82	10.95	7.87	24.79	17.18	12.36	103.05	95.73	77.97
34.96	16.87	11.41	8.47	26.24	18.28	13.20	106.16	98.85	80.92
40.33	18.94	12.94	9.67	29.02	20.38	14.84	111.71	104.44	86.32
45.70	20.93	14.43	10.84	31.65	22.39	16.41	116.54	109.34	91.09
51.05	22.84	15.86	11.98	34.12	24.31	17.91	120.75	113.65	95.37
56.42	24.69	17.23	13.08	36.49	26.15	19.35	124.54	117.51	99.24
69.85	29.10	20.53	15.78	42.00	30.47	22.82	132.48	125.66	107.51
75.23	30.76	21.72	16.79	44.05	32.11	24.11	135.23		110.38
80.62	32.39	22.92	17.80	46.03	33.70	25.39	137.80		113.03
85.98	33.97	24.19	18.78	47.93	35.23	26.62	140.13		115.47
91.29	35.48	25.36	19.72	49.76	36.70	27.82	142.29		117.74
96.69	37.00	26.53	20.68	51.56	38.16	29.01			119.92
99.50		27.12	21.17	52.48	38.92	29.61			121.02

Table 5. Parameters for Fitting Viral Eq 1 to the Experimental Data, σ (rms Error), and Calculated Q^0_{st}

system	$10^{-3}a_0$	a_1	10^2a_2	10^3a_3	b_0	σ/(cm^3·g^{-1})	Q^0_{st}/(kJ·mol^{-1})
G/CH_4	−2.83	6.58	−1.75		10.17	0.18	23.5
G/CF_4	−3.41	8.01	−0.76		11.50	0.43	28.3
G/SF_6	−4.99	18.36	−19.7	1.76	13.76	0.54	41.5
W/CH_4	−2.34	11.7	−16.6		9.69	0.04	19.5
W/CF_4	−2.54	7.36			10.10	0.33	21.1
W/SF_6	−3.67	20.6	−31.2	2.32	11.22	0.12	30.5
M/CH_4	−2.18	3.28			8.67	0.33	18.2
M/CF_4	−2.64	7.41	−9.14	0.71	9.61	0.09	21.9
M/SF_6	−3.83	8.26	−5.41	0.22	11.18	0.67	31.8

quantity of adsorption, can be directly derived from eq 1, and it takes the following form:

$$Q_{st} = -R\left(\frac{\partial \ln p}{\partial(1/T)}\right)_v = -R\sum_{i=0}^{m} a_i v^i \qquad (7)$$

The isosteric enthalpy of adsorption at zero coverage, Q^0, often used as a parameter to characterize adsorption energetics, is given by

$$Q^0 = -Ra_0 \qquad (8)$$

The values of Q^0 for all systems studied are also presented in Table 5. The variations of the isosteric enthalpies of adsorption for each carbon versus the amount adsorbed calculated from eq 7 for the case of CF_4 are shown in Figure 3. The highest enthalpy of adsorption for Carbosieve G indicates the presence of small pores of sizes comparable to the adsorbate molecular diameter. In such pores the adsorption potential is strongly enhanced. The enthalpies of adsorption on Maxsorb and Westvaco carbons are similar to each other, indicating that these carbons have pores in the same range of sizes.

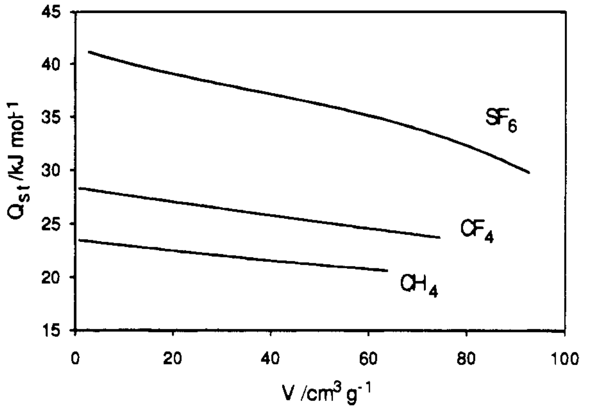

Figure 4. Isosteric enthalpies of adsorption of CH_4, CF_4, and SF_6 on Carbosieve G.

The variation of Q_{st} versus v for different gases on Carbosieve G is compared in Figure 4. The monotonic decrease of Q_{st} reflects the energetic heterogeneity of this carbon. The values of Q_{st} increase in the order of CH_4, CF_4, and SF_6 which corresponds to the order of adsorption potentials for these molecules. Consequently, values of the isosteric enthalpy at zero coverage, Q^0 (Table 5), show the same trend.

In conclusion, it was shown that adsorption data measured for gases near their boiling point can provide important information about the microstructure of microporous materials. This information can be extracted using a simple thermodynamic approach in which a virial-like description of the results has been used. These results may also be used for more sophisticated analyses involving detailed adsorption models. In our laboratory, additional analyses are in progress (Jagiełło *et al.*, 1995; Bandosz *et al.*, 1995).

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